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SOLID STATE RADIOGRAPHIC  
IMAGE AMPLIFIERS  
Final Report: Part C

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16. ABSTRACT  <p>This report describes the third part of the program for the development of solid state radiographic amplifiers. The main goal of this program was to improve the contrast sensitivity of the radiographic amplifiers, both the storage type and non-storage type, improve their absolute sensitivity and the reproducibility of fabrication.</p> <p>The required 2-2T quality level, per MIL-STD-453, was reached with the radiographic storage screen. The sensitivity threshold was 100 to 200 mR with 45 to 100 kV filtered x-rays. The quality level of the radiographic amplifier screen (without storage) was 4-4T (for a 6mm (0.25") thick aluminum specimen, a 1mm (0.040") diameter hole in a 0.25mm (0.010") thick penetrameter was detected). Its sensitivity threshold was 2 to 6 mR/min. The developed radiographic screens are applicable for uses in non-destructive testing.</p>			
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## SUMMARY

The work in this third part of the program on solid state radiographic amplifiers was directed toward the improvement of contrast sensitivity, absolute sensitivity, and reproducibility of both storage and non-storage type radiographic amplifiers.

Most of the effort was devoted to work on the radiographic storage screen (R.S.S.). The main goal, a quality level of 2-2T, or detection of a 0.5mm (0.020") diameter hole in a 0.12mm (0.005") thick penetrometer for 6mm (0.25") thick aluminum specimens, according to MIL-STD-453, was reached. The absolute sensitivity (speed) of the storage panel was between those of Kodak type M and type R films. The sensitivity threshold (the x-ray dose needed to double the background brightness) was between 100 to 200 mR with 45kV to 100 kV filtered x-rays. A dose of about 9R was needed for displaying the 2% penetrometer on a 6mm (0.25") thick aluminum with 45 kV x-rays filtered through 3mm (0.12") Al. The dose on the storage screen itself was less than 1R in this case.

The reproducibility of the storage screen fabrication was improved.

The non-storage type radiographic amplifier screen (R.A.S.) reached the quality level of 4-4T (a 1mm (0.040") diameter hole in a 0.25mm (0.010") thick penetrometer on a 6mm (0.25") thick aluminum specimen was detected). The threshold radiation level (to two times background brightness) of these screens was about 2 to 6mR/min intensity with filtered 45 kV to 100 kV x-rays. The 6mm (0.25") thick Al 4% penetrometer was clearly visible with an intensity of 100mR/min (45 kV - no filter). The picture quality (reduction of graininess) and the reproducibility of fabrication of these panels was improved.

Eight 20cm x 25cm (8" x 10") size radiographic storage screens, one 23cm x 25cm (9" x 10") size, and several 7.5cm x 7.5cm (3" x 3") size radiographic amplifier screens, and a power supply for driving and erasing the R.S.S. were delivered to MSFC.\*

The R.A.S. can withstand and function in a high vacuum atmosphere, but for the proper working of the R.S.S. oxygen is important. Therefore, for possible use in space, the storage screen should be sealed in an oxygen atmosphere or with some other material which is capable of delivering oxygen to the ZnO layer. Further work would be needed to achieve this goal.

The further development of flexible R.S.S. is also recommended, as detailed in reference 2.

\*Marshall Space Flight Center

## SECTION I

### INTRODUCTION

This report gives an account on the work performed on Contract No. NAS8-25678 from March 19, 1970 to May 31, 1971. This is the third part of the program on Solid State Radiographic Amplifiers, which started on July 1, 1967 under Contract No. NAS8-21206. Final reports on the first two parts were edited on May 1968<sup>(1)</sup> and on November 1969<sup>(2)</sup>.

During the first two parts of the program, x-ray and visible light-sensitive storage and non-storage type solid state image amplifier panels were developed for use in industrial nondestructive testing with the view of possible applications in future space flights. The storage-type radiographic panels were intended to substitute for radiographic films, and the non-storage types to substitute for fluoroscopic screens. Photoconductor-electroluminescent (PC-EL) sandwich type construction was the basic design used in this work. Cadmium selenide-cadmium sulfide (CdSe-CdS) was the sensor photoconductor for the non-storage type and zinc oxide (ZnO) for the storage-type panels. Though the characteristics of these panels met the requirements for some applications, with continued work the possibility of further improvements was expected, extending the field of applications.

The main goal of the third part of this program was to reach the 2-2T quality level (MIL-STD-453) with the storage-type radiographic amplifiers and approach it on the non-storage type panels. Other goals were to improve absolute sensitivity and reproducibility of fabrication.

The 2-2T quality level was reached on the radiographic storage screen. Its other characteristics met the requirements for industrial nondestructive testing applications so that it is a good substitute for radiographic films, diminishing the expense and increasing the speed (no development, immediate viewing) of radiographic work.

The developed non-storage type panels may be used in many applications where the fluoroscopic screen, x-ray intensifier tube or TV camera system is now used, because of their following characteristics (compared to the DuPont CB-2 screen): (1) three times higher resolution; (2) two to six times higher contrast; (3) ten to fifty times higher brightness in a given x-ray intensity range; (4) light-weight and easy to operate.

The work on this program was performed at the Electronic Tube Division of the Westinghouse Electric Corporation, Elmira, New York by R. Bentley and C. A. Lepkowski, with Z. Szepesi as project Engineer. Managerial supervision

(1)Reference to superscript numbers to be found in Section 8.

was provided by G. W. Goetze, followed by D. D. Doughty, R. A. Shaffer and G. B. Conger.

The project engineer of this contract at the Marshall Space Flight Center was J. B. Beal.

## SECTION 2

### OBJECTIVES

The objectives of the contract were to improve the characteristics of the storage and non-storage type radiographic amplifier as specified herein and deliver a given number of them to MSFC.

#### 2.1 Storage Type Panels

Eight 20cm x 25cm (8" x 10") size radiographic storage screens were to be fabricated. The operational parameters of these panels should be optimized for high gain, visibility in ambient light levels of 108-216 lux (10-20 foot candles) and resolution higher than 4 line pairs/mm (200 TV lines/inch). As the optimum goal, radiographic quality levels of these panels should reach that of 2-2T, as defined by MIL-STD-453, using 6mm (0.25") thick aluminum plates with low energy gamma and x-ray sources not exceeding 60 keV. Secondary parameters are: short erasure time and usable storage time exceeding ten minutes. The weight and thickness of a panel should not exceed two pounds and 12.7mm (0.5"), respectively.

Consideration shall be given to (1) a two-stage cascaded construction, combining a storage panel with a light amplifier panel; (2) application of dark field EL material (available from Sigmatron, Inc.).

#### 2.2 Non-Storage Type Panels

One 23cm x 25cm (9" x 10") size radiographic amplifier screen, non-storage type, was to be fabricated. The operational parameters of this amplifier panel were to be optimized for high resolution and gain. Image resolution shall be higher than 4 line pairs/mm (200 TV lines per inch), if possible. It was to be compatible with low energy radiation sources, not exceeding 60 keV. Efforts were to be made to lower input radiation requirements (high absolute sensitivity). The contrast sensitivity should also be high, approaching the quality level of 2-2T (MIL-STD-453). Electrical requirements for panel operation were to be in the range of 100 to 200 volts, 60 to 400 Hz.

## SECTION 3

### METHOD OF APPROACH

The basic approach for satisfying the requirements of the contract was the same as in the previous part of the program: by photoconductor-electroluminescent (PC-EL) type image amplifiers. Continuous layer, sandwich type construction was used, where the PC and EL layers were sandwiched between two conductive electrodes. The history and working principle of this construction is described in Reference 2, on pages 3 to 11.

#### 3.1 Radiographic Storage Screens (R.S.S.)

To improve the contrast sensitivity and meet the specifications on the storage panel (quality level of 2-2T), several approaches were considered.

Beside improving the contrast (gamma) of the R.S.S. itself, the characteristics of the human eye for detecting small brightness differences have to be considered. Experiments proved<sup>(3)</sup> that the threshold of detection depends on the apparent size of the object (more precisely on the viewing angle), on brightness and wavelength of the light. The value of this contrast threshold is lower when the viewing angle or the brightness are increased, and when the wavelength is decreased. Also, the graininess of the panel has a large effect on the detectability of small objects. Consequently, it was planned to:

- (1a) Change the doping and processing of the ZnO powder to increase the gamma of the photoconductor transfer characteristic.
- (1b) Find optimum construction parameters, such as thickness, plastic to powder ratio, ball milling, etc., for highest contrast of the panel.
- (1c) Incorporate a non-linear layer in the sandwich to obtain higher gamma.
- (1d) Use EL layers (powder or evaporated) with high discrimination ratio.\*
- (2) Shorten the wavelength of the output light, using blue EL phosphor.
- (3) Increase brightness. One way to realize this could be by increasing the thicknesses and using higher voltages on the storage panel. Another way to reach higher brightness could be by merging two amplifier panels in a cascade arrangement, i.e., combining a storage panel with a light amplifier panel.

\*Brightness ratio of the EL panel with a given voltage to that with half of the voltage.

- (4) Decrease graininess by changing processing parameters of the ZnO powder.

A series of experiments, changing the important parameters one at a time, were planned to find the optimum combination for highest contrast so that secondary characteristics, such as absolute sensitivity (speed), resolution and erasure time, would still be acceptable and satisfy requirements.

### 3.2 Radiographic Amplifier Screens (R.A.S.)

To improve the contrast and absolute sensitivity of the non-storage type panels, a series of experiments were planned where the doping and processing parameters of the photoconductive powder would be changed. The resolution did not seem to be a problem, but the graininess had to be reduced and better reproducibility attained.

## SECTION 4

### FABRICATION TECHNIQUE

Both the storage and non-storage type radiographic screens are of the same general construction. However, differences in many details exist because the photoconductive layer in the storage screen is a plastic-embedded structure and in the non-storage screen it is a sintered powder.

#### 4.1 Radiographic Storage Screen

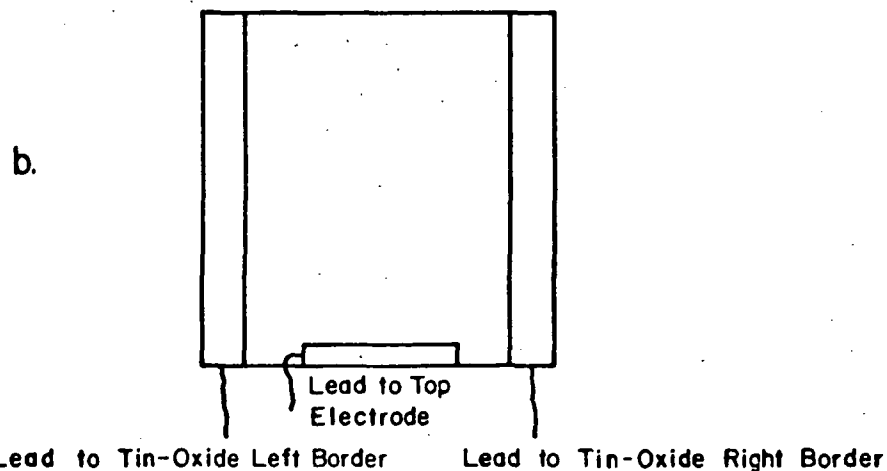
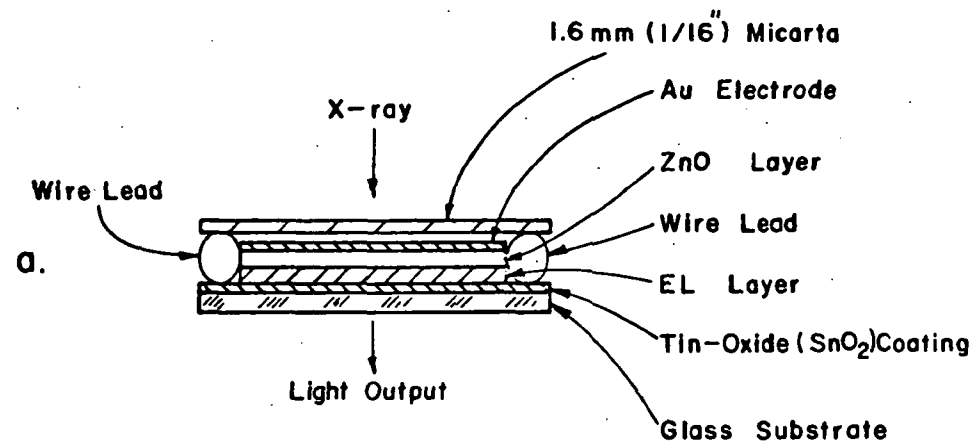
The construction of the R.S.S. is shown in Figure 1. The detailed step-by-step processing is described in Appendix A. All the steps, excepting a few details, are the same as specified in the previous (second) part of this program (Reference 2, Section 4.1.1 and Appendix D).

#### 4.2 Radiographic Amplifier Screen

The construction of the R.A.S. is shown in Figure 2. Fabrication details are described in Appendix B. The processing steps are almost the same as specified in the previous part of this program (Reference 2, Section 4.1.3 and Appendix E).

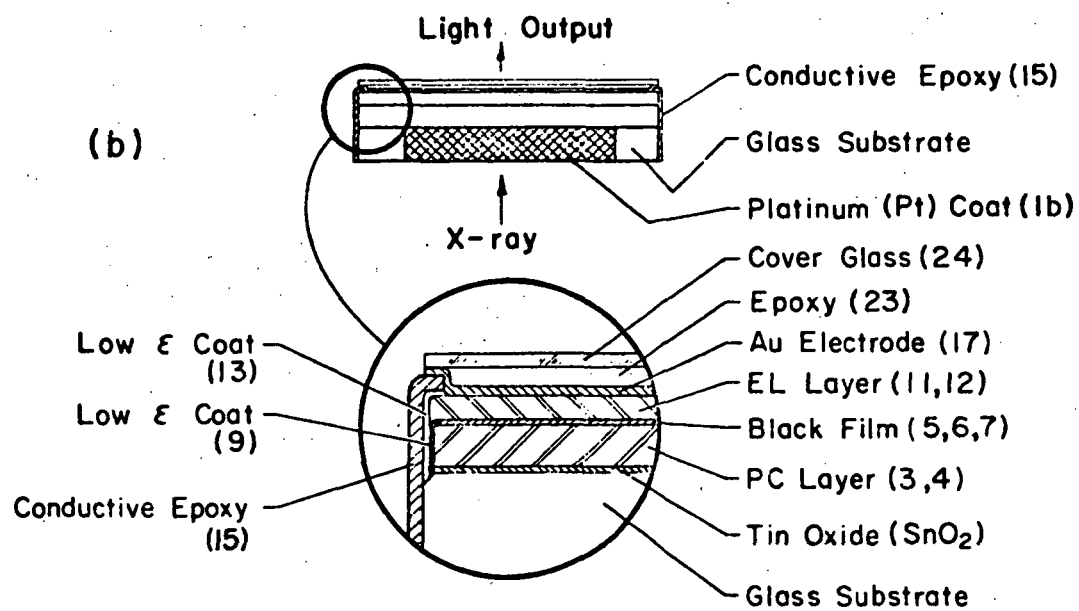
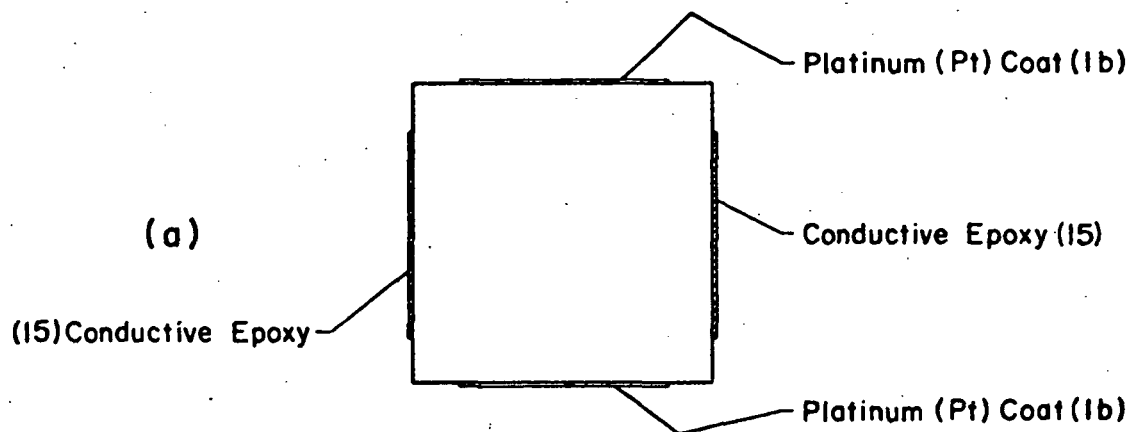
#### 4.3 Power Supply for R.S.S.

A power supply, containing the driving voltage and erase power for the R.S.S. was constructed. The driving voltage was obtained through a 1:4 transformer from the 60 Hz power line, which could be set between 0 and 500 volts by a variable transformer. The erasing power was controlled by a commercial solid state light dimmer. The circuit diagram of the power supply is shown in Figure 3.



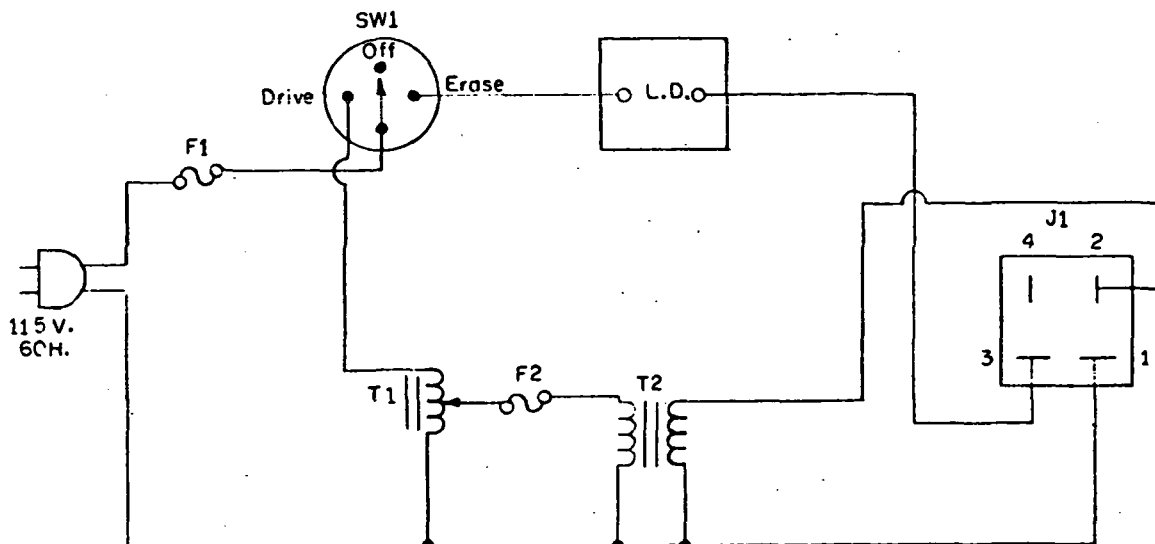
(a) cross section (b) top view

Figure 1. Construction of Radiographic Storage Screen



(a) Top View; (b) Side View and Cross Section  
Number in Parentheses refer to Appendix B, Part I.

Figure 2. Construction of Radiographic Amplifier Screen



Power Supply 25678-1

IONS		ELECTRICAL BILL OF MATERIAL				
	ITEM NO.	AMOUNT REQUIRED	SYMBOL	MANUFACTURER	CATALOG OR STYLE NO.	DESCRIPTION
B-E	1	1	F1	Littelfuse	342014	5A fuse
	2	1	F2	Littelfuse	342014	0.5A fuse
	3	1	T1	SuperiorElec	10B	Variable Transformer Powerstat
	4	1	T2	Stancor	PC8418	Power Transformer
	5	1	L.D.	Ideal Indus	56-0010 NAE04250	Light Dimmer
	6	1	J1	Clinch-Jones	S-304DB	Socket
	7	1	SW1	Cutler-Hammer	7011K1	Rotary Switch
	8	1	---	Zero Mfg. Co.	ZC-7060	Instrument Case
				USED ON SUB-ASSEMBLY	EQUIPMENT ENGINEERING ELECTRONIC TUBE DIVISION HORSEHEADS, N. Y. WESTINGHOUSE ELECTRIC CORPORATION	MACHINE Power Supply 25678-1 B-E

Figure 3. Circuit Diagram and Bill of Materials of Power Supply for R.S.S.

## SECTION 5

### EXPERIMENTAL RESULTS

The larger part of the program was conducted toward meeting the contrast sensitivity requirement (quality level 2-2T) for the radiographic storage screen. A smaller effort was devoted to the improvement of contrast and gain of the radiographic amplifier screen.

For the evaluation of the various experiments, the numerical measurement of the contrast (gamma) was very important. Test equipment was built for this purpose, which is described in the following paragraph.

#### 5.1 Contrast Measuring Equipment

The contrast sensitivity of the radiographic panel is determined by the non-linearity of its input-output (transfer) characteristics. The relation between input x-ray dose ( $L = I \times t$ , where  $I$  is intensity,  $t$  is exposure time) and output brightness ( $B$ ) can be described in the practical useful region of the transfer characteristic by the following equation:

$$B = B_1 L^{\gamma}$$

Here,  $B_1$  is a constant and  $\gamma$  (gamma) is the measure of non-linearity, which determines the contrast sensitivity. If we take the logarithm of the two sides:

$$\log B = \log B_1 + \gamma \log L$$

we see that  $\gamma$  is the slope of the transfer characteristic drawn on a double logarithmic paper. This is the same interpretation as the characteristic curve (H & D curve) of photographic films, representing density (logarithm of reciprocal transmission) against logarithm of light intensity-exposure time product.

For an objective evaluation of the contrast sensitivity of radiographic panels, the determination of gamma was necessary. At the beginning of the work, the brightness was recorded as a function of time when the panel was exposed to x-rays of given intensity. From this curve,  $\log L$  and  $\log B$  were calculated and the characteristic curve was traced, from where the gamma could be read.

To simplify the process, logarithmic amplifiers were connected to both the brightness and time axes, and the characteristic curve was recorded this way in a double logarithmic scale with an X-Y recorder. The block diagram of the recording set-up is shown on Figure 4. The brightness sensing cell

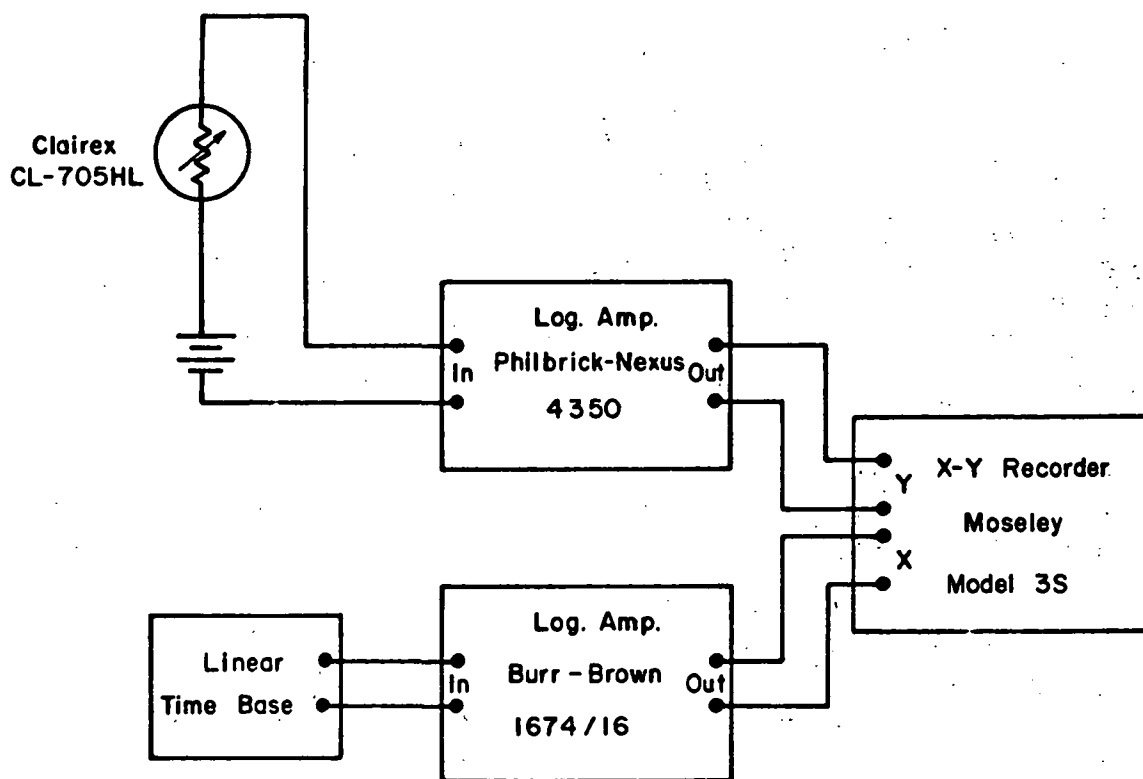


Figure 4. Block Diagram of Characteristic Curve Recorder

at the beginning of the work was a CdS photoconductive cell (Clairex CL-705HL), which had nearly linear current vs light intensity characteristic. A Philbrick-Nexus type 4350 logarithmic amplifier was connected to the photocell, followed by the Y-axis of a Moseley Model 35 recorder. Similarly, to the X-axis of the recorder, the output of a Burr-Brown type 1674/16 logarithmic amplifier was connected, which was driven by a linear time-scale. A button on the timing circuit, pushed down at the same time as the x-ray voltage was connected, started the recording.

It was found that the deviations from linearity of the CdS photocell caused appreciable errors in the determination of the gamma. Therefore, the CdS photocell was replaced by a silicon-diode (UDT-PIN-3D), followed by an amplifier. The circuit diagram of the Si-diode amplifier is shown in Figure 5.

The Si-diode has, beside its exact linear response (short circuit current) in seven decades of input light, other advantages compared to the CdS photocell. It is faster, even at the lowest light levels of  $10^{-8}$  Lamberts (about  $10^{-5}$  fL), by a factor of more than  $10^{-5}$ . It does not have a previous exposure effect like the CdS photocell, i.e., the measured current is not affected by the previous illumination.

The above-described equipment functioned well, and simplified the evaluation of the contrast sensitivity of the experimental storage panels.

## 5.2 Radiographic Storage Screen

As it was discussed in Section 3 of this report, the following goals directed the experimental work on the R.S.S.:

1. Increase the gamma of the storage screen itself.
2. Use light output with shorter wavelength.
3. Increase the brightness.
4. Decrease graininess of the image.

### 5.2.1 Experiments for Increased Gamma.

In order to increase the gamma of the R.S.S., the following possibilities were considered:

1. Changing the doping and processing of the ZnO powder.
2. Changing the construction parameters of the panel, such as thickness of the layers, plastic to powder ratio, and ball milling parameters.
3. Incorporating a non-linear (superlinear current vs voltage) layer in the sandwich.
4. Using EL layers with high discrimination ratio.

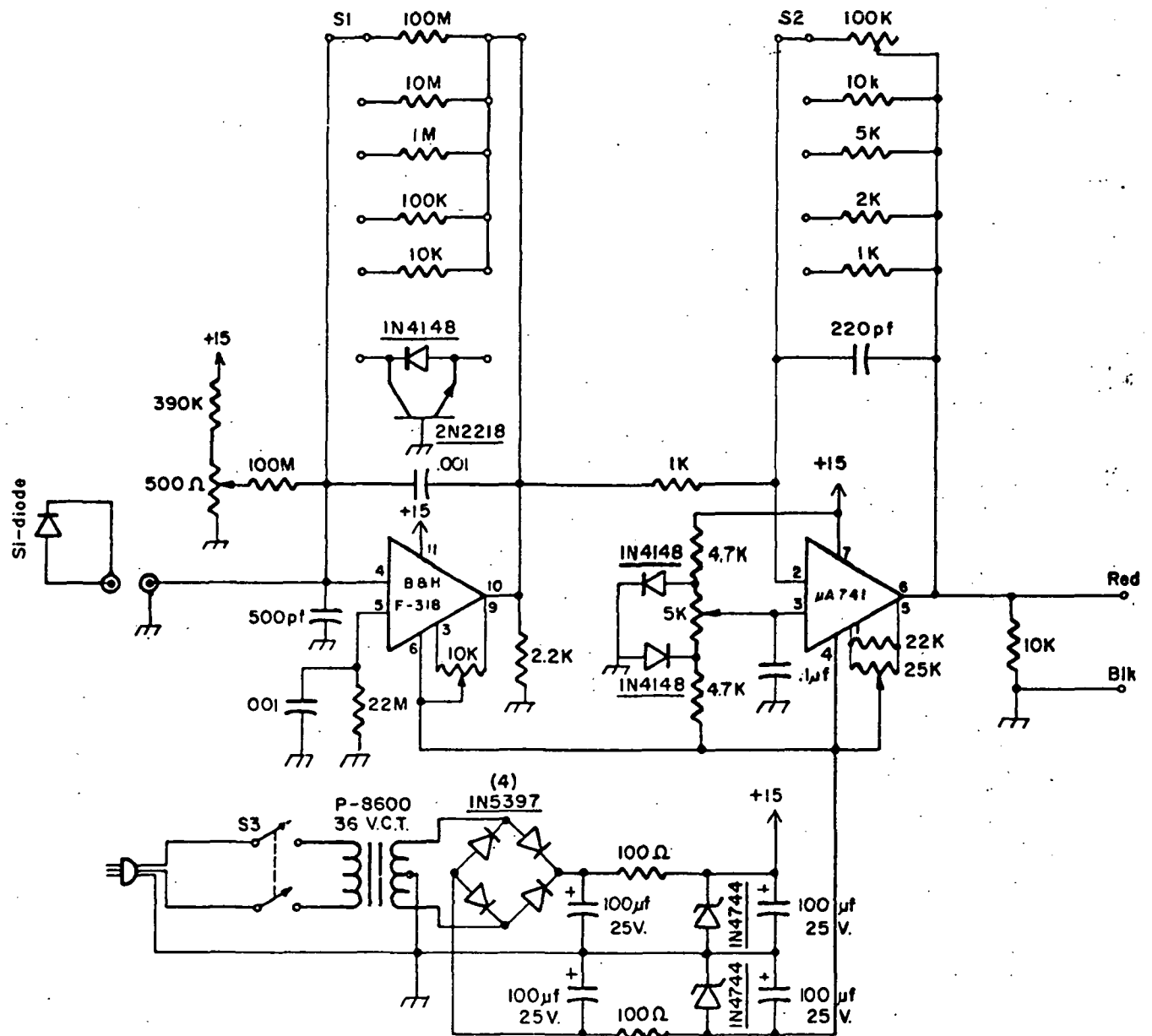


Figure 5. Circuit Diagram of Si-diode Amplifier

#### 5.2.1.1 Preparation of ZnO powder

The characteristics of the ZnO powder strongly depend on the doping of the powder, which is a function of the impurities added and the processing parameters, such as baking temperature and time, and the ambient atmosphere of the furnace. In the second part of the program, it was found that about 0.088% by weight  $\text{Na}_2\text{SO}_4$ , mixed with the ZnO powder and baked in slowly flowing air at  $950^\circ\text{C}$  for two hours, gave the best characteristics.

Experiments were made with other dopants, such as  $\text{PbO}$ ,  $\text{FeO}_3$ ,  $\text{PbCl}_2$  and  $\text{MgCl}_2$ . Also, the baking temperature and time were changed from  $900^\circ\text{C}$  to  $1200^\circ\text{C}$  and from 60 minutes to 300 minutes, respectively. Baking was done in stagnant air, in an air flow and in oxygen flow. The results of these experiments were:

- a. Baking in stagnant air resulted in high dark currents.
- b. Too short and too long baking time diminished the sensitivity and contrast.
- c. Too high temperature resulted in large grains, too low temperature in low sensitivity and contrast.
- d. Oxygen atmosphere did not give any improvements over air atmosphere.
- e. The chlorine doping resulted in finer grain.
- f. The highest contrast and sensitivity was reached with a combination of  $\text{Na}_2\text{SO}_4$  and  $\text{PbCl}_2$  doping and resulted in the detection of 2T holes on the 2% penetrometer for 6mm (0.25") thick aluminum, using 45 kV x-ray tube voltage.

The best results were reached with a 0.1%  $\text{PbCl}_2$  and 0.088%  $\text{Na}_2\text{SO}_4$  doping, a baking temperature of  $1000^\circ\text{C}$  and a 2-hour baking time. It was found later that the reproducibility of this processing was poor. The changing humidity content of the airflow was suspected and a new series of experiments were conducted, using a liquid nitrogen cooled water-trap in the air flow. The best powder produced in this series of experiments contained 0.075%  $\text{PbCl}_2$  and 0.125%  $\text{Na}_2\text{SO}_4$ , and was baked at  $950^\circ\text{C}$  for 2 hours in a slow dry airflow. This schedule produced good reproducible characteristics, and all delivered storage panels were made with this kind of ZnO powder.

#### 5.2.1.2 Construction Parameters

Several series of experiments were carried out, changing the construction parameters of the R.S.S. The thickness of the ZnO layer was varied between 0.2 and 0.5mm (0.008" and 0.020"), the plastic (DC804 silicone resin) ratio was between 2% and 12%, the ball milling time was between zero and 20 hours, and the viscosity of the coating mixture was changed (one parameter per series of experiments).

The evaluation of these experiments resulted in the following general conclusions:

- a. Thinner layers caused:
  - . higher gamma
  - . higher background brightness

- shorter storage time
- b. Lower plastic content caused:
  - no change in sensitivity and gamma
  - lower background brightness
  - shorter storage time
- c. Longer ball milling caused:
  - finer graininess
  - lower absolute sensitivity
  - lower background brightness
  - lower gamma
  - shorter storage time

The viscosity of the coating mixture seemed to cause an indirect effect: mixtures with lower viscosity had more effective ball milling and showed characteristics similar to longer milled mixtures. Lower viscosity was advantageous for getting more uniform coatings without background line structures, which were inevitable when the viscosity was high. Lower background brightness meant that the dark current of these panels was low; with this, the absolute sensitivity was low. One had to use higher voltages on these panels for acceptable sensitivity. However, if these voltages were excessively high (600 to 800 volts), these panels were rejected.

Similar problems arose when a new batch of the DC-804 silicone resin was obtained and used. The required activation voltage was around 800 volts; therefore, the thickness of the ZnO layer was to be decreased, and the plastic concentration increased so that 600 volts or less would be adequate for the proper working of the R.S.S.

The final compromise of construction parameters for the ZnO layer was 0.44mm (0.0175") thickness, 8 to 10% DC-804, and 45 to 50 minute ball milling of an easy-flowing powder mixture.

The solvent used before the  $PbCl_2$  doping of the ZnO powder was butyl carbitol. Good, uniform layers were easily coated on top of the EL layer with these powder mixtures. However, when the  $PbCl_2$  doped powders were introduced, the deposited ZnO layers and the output image of the storage panel showed a mottled, non-uniform structure. Experiments conducted with different solvents lead to the final use of iso-amyl-alcohol with a low amount of butyl carbitol (see Appendix A).

#### 5.2.1.3 Non-Linear Layer

It was thought that incorporating a non-linear (NL) layer in the sandwich construction would increase the contrast sensitivity of the storage panel, similar to the NL layer and EL layer combination which results in high slope and discrimination ratio of the EL characteristics.

NL layers were prepared from chlorine-doped CdS powder. Electronically pure cadmium sulfide powder, mixed with different amounts of cadmium chloride (0.05, 0.16 and 0.5%), was baked in nitrogen atmosphere. These powders, embedded in a low dielectric plastic, showed good stability and appropriate resistivity. Combined with an EL layer (EL-NL sandwich), the discrimination ratio increased about ten times. However, built in the radiographic amplifier structure (PC-NL-EL construction), the contrast of this combination panel did not improve. The panel had lower resolution and lower putput, probably due to the increased thickness of this construction.

After these preliminary experiments, the work with NL layers stopped, because the 2-2T quality level was reached with the simple PC-EL construction.

#### 5.2.1.4 High Discrimination Ratio EL Layers

Some effort was directed in 1958 and 1959 toward the production of EL phosphor powders with high discrimination ratio<sup>(4)</sup>. The best phosphor resulted from this study had a discrimination ratio of about 50 at  $5.4 \times 10^{-3}$  Lamberts (5 fL) and 400 Hz. Since the discrimination ratio depends on the brightness and on the construction parameters of the cell, it was not expected to reach this high value under the restricted conditions of a PC-EL panel construction. Also, the phosphor powder made 12 years ago, had deteriorated, and the radiographic storage screens fabricated with this special phosphor did not show appreciable improvements in contrast sensitivity.

Another type of EL construction known to offer high discrimination ratio was the evaporated film. The DC driven EL evaporated films show very high discrimination ratios, which increase with brightness, contrary to the deposited powder EL layers. Some experiments were made with small evaporated DC-EL films which were prepared about ten years ago. The characteristics of these films were not very stable and had deteriorated, thus no improved contrast sensitivity was detected on the storage screens fabricated with them.

A recently made "high contrast" EL film sample was obtained from the Sigmatron Corporation. The "high contrast" here implies another characteristic: the contrast between illuminated and non-illuminated areas does not deteriorate in high ambient illumination. The discrimination ratios of these evaporated films, which are used with AC voltage, were somewhat higher than those of the powder layers, though not as high as those of direct current EL films.

A radiographic storage screen, built on the Sigmatron EL film, did not show improved contrast sensitivity. It is possible that some improvement could be reached with optimum construction parameters of the storage screen, but the activity in this direction was discontinued because of lack of time and EL samples.

#### 5.2.2 Blue Output Light

It was found (see reference 3) that the contrast threshold of the eye is lower at shorter wavelength illumination. The standard EL phosphor

generally used, which has the best brightness and life (maintenance) characteristics, has an emission maximum at around 530 nm. It was expected that a blue EL phosphor, which has an emission maximum at around 490 nm could bring in some improvement in the contrast sensitivity of the radiographic screen. However, panels prepared with such a blue phosphor did not show appreciable improvement, probably due to the lower brightness of the blue phosphor.

### 5.2.3 Increased Brightness Output

Experiments carried out by Lamar and others showed an improved contrast detectability (lower contrast threshold) with increased brightness. (3) We conducted experiments for increased brightness of the radiographic storage screen along two lines: (1) improving the brightness of the simple PC-EL construction by changing electrical and construction parameters; (2) increasing the brightness by an intensifier panel (also a PC-EL construction) cascading the two panels together.

#### 5.2.3.1 Single Units

Increasing the voltage of a given PC-EL panel increases the output brightness. However, the gamma diminishes (because the discrimination ratio of the EL layer diminishes), and the contrast sensitivity of the panel decreases.

Increasing the driving frequency also increases the brightness. Although the gamma does not diminish, practically no improvement in contrast sensitivity was noticed.

Constructing the panel with a thicker EL layer increases the brightness, though higher driving voltage has to be used. Visual observation on such panels, however, did not show detectable improvement in the contrast sensitivity.

#### 5.2.3.2 Cascaded Amplifiers

A straightforward way to increase the brightness and still keep the gamma undiminished or even increased is by cascading two amplifier panels. (5) Two different constructions can be thought of:

- (1) First panel is a R.S.S., the second a light sensitive image amplifier.
- (2) First panel is a R.A.S., the second a light sensitive storage screen.

The first approach is not feasible with the fabrication method used, because one can not build above the plastic embedded layers of the R.S.S. a light sensitive image amplifier, which needs 500°C sintering temperature for its PC layer. Building separately the two panels, the output EL layer of the first and the input PC layer of the second panel are separated by

the substrate glass of the second panel, causing an unacceptable deterioration in the resolution. If plastic embedded PC powder could give acceptable characteristics, it would be possible to follow this approach, but we did not succeed in building such image intensifier panels with satisfactory characteristics.

The second approach can be realized, if one has a light sensitive storage screen. Such panels were developed in the second part of the work program (see reference 2, pages 17, 28 and 48). However, the sensitivity (photographic speed) of these panels was only ASA  $10^{-2}$  (meter-candle-second)<sup>-1</sup> at the beginning of this work program and was improved by a factor of about 5 later. Still a sensitivity of ASA  $5 \times 10^{-2}$  was not enough for the cascaded construction, and time was not available for more effort in this approach.

#### 5.2.4 Graininess

To detect the small 0.5mm (0.020") diameter hole on the 2% penetrometer, the structure of the panel should not show coarse, grainy structure. The sensitized ZnO powders generally resulted in a storage screen with disturbingly large grains. However, the grain size was lower if the sensitizing temperature of the powder was lower. But too low temperature caused lower absolute sensitivity (speed) and lower contrast. After evaluating the results of a series of experiments, a compromised baking temperature of 950°C was chosen.

Powders baked at 950°C still presented some graininess. Ball milling of the sensitized powder was tried for decreasing the grainy structure. It was found that longer ball milling diminished the graininess, but also diminished the speed, contrast and storage time. For optimum characteristics ball milling time of 40 to 60 minutes, the time depending on the viscosity and on the quantity of the mixture, was chosen.

#### 5.2.5 Characteristics of R.S.S.

The characteristic curves (logarithm of brightness vs logarithm of x-ray dose) of the R.S.S. were recorded with the set-up described in paragraph 5.1. The standard x-ray tube voltage for these recordings was 45 kV, current 2mA and tube-panel distance 91cm (36") giving an intensity of 7 R/min through a plastic sheet, which held the panel. The voltage on the R.S.S. was set to a value where the background brightness before exposure was about  $5.4 \times 10^{-6}$  Lamberts ( $5 \times 10^{-3}$  fL). Figure 6 shows such a recording. The length of the exposure was 60 seconds (the rising part of curve 1) after which the brightness started to decay. Curve 2 is the continuation of the decay curve with a 5 times slower time-scale.

Measurements proved that the reciprocity law was valid\* unless the intensity was too low and exposure times much longer than the storage time were needed.

\*The reciprocity law states that when one changes the intensity of the x-ray (I) without changing other parameters, the necessary exposure time (t) for reaching the same output brightness is proportional to the reciprocal of the intensity, i.e.:  $I \times t = \text{const.}$

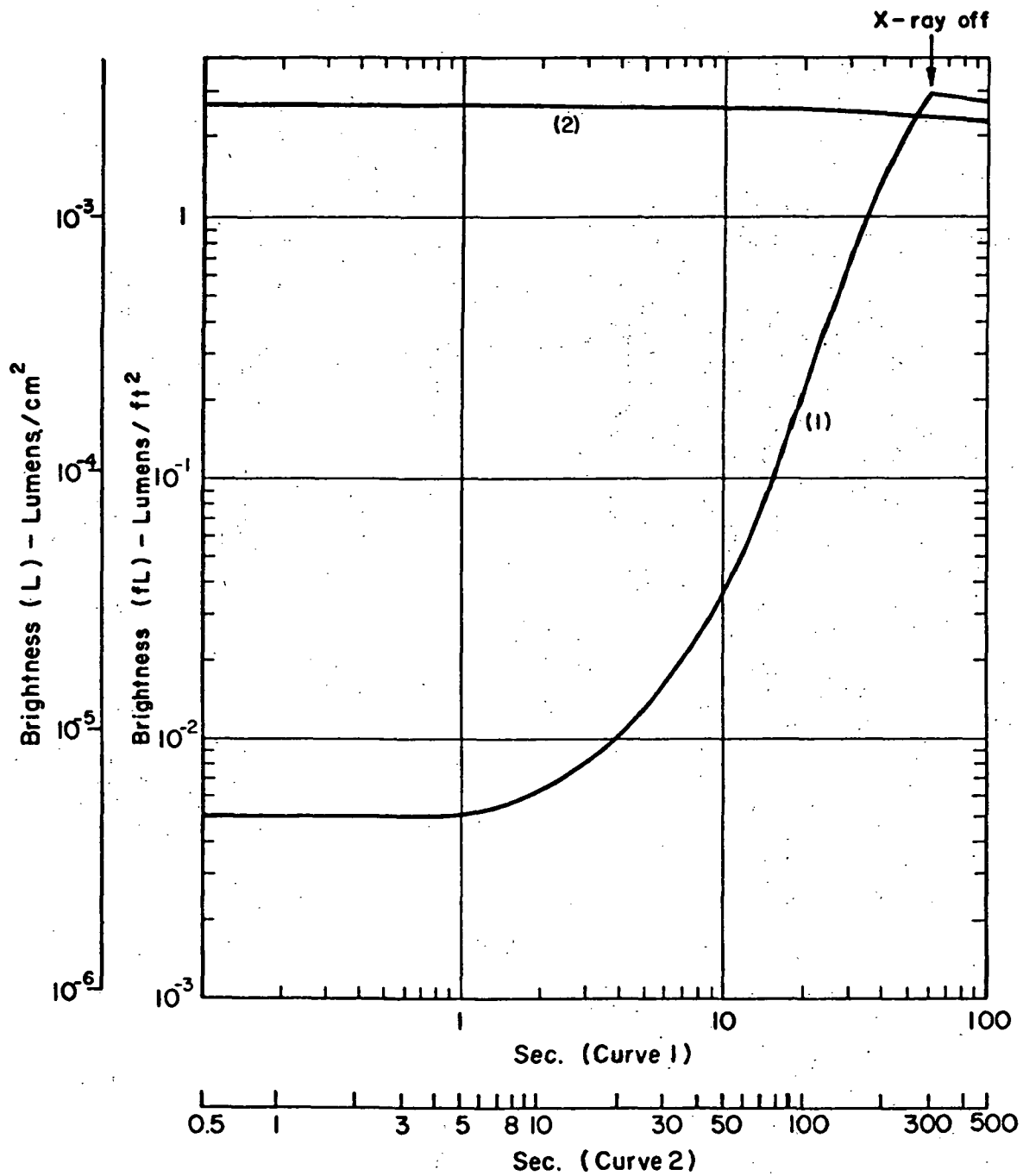


Figure 6. Characteristic Curve of an Average R.S.S.

The resolution of the R.S.S. was about 8 line pairs per millimeter (400 TV lines per inch).

The storage time was between 10 and 60 minutes, defined as the time where the brightness drops to one third. It was not a constant value for a given storage screen. It was longer, when the panel was exposed to higher doses (higher output brightness), and also, when the driving voltage on the panel was higher.

The explanation of these phenomena is thought to be the following: (1) When the panel is exposed to higher x-ray doses, more traps are filled up with electrons, which will be available during the decay period, giving longer storage time. (2) Higher voltage brings higher electric field on the ZnO layer which results in the extraction of more electrons from deeper holes.

The truth of these assumptions could be proved experimentally, but limited time did not allow this.

The resolution and the contrast did not deteriorate noticeably during the storage time.

The storage time was much longer when the driving voltage was not connected to the panel. This way, reasonably good pictures could be displayed even several days after the exposure. Cooling down the panel probably could increase the storage time, but this hypothesis was not proved experimentally.

The driving voltage has an appreciable effect on the value of the gamma of the R.S.S. Figure 7 shows two characteristic curves recorded with different voltages. As we see, the speed of the screen diminishes with the voltage, but the gamma increases. Practically, this means that for obtaining higher contrast sensitivity, longer exposure and lower driving voltage should be used.

The erasure of the R.S.S. needs heating of the ZnO layer to around 100°C for 3 to 20 minutes, which could be achieved by an infrared lamp illumination or placing the panel in a furnace. However, the best way was to heat the tin oxide coating of the glass substrate electrically. All the panels finished and delivered had electric bus-bar connections on opposite borders of the tin oxide coating, which served for the application of the electric heating power. About 0.5 Watt/cm<sup>2</sup> (3 Watt/square inch) heated the ZnO layer to about 130°C in 3 or 4 minutes, and resulted in a good erasure. The cooling needed about 5 minutes, when the panel was placed on a large aluminum plate and a fan moved the air around it.

For good erasing, the ZnO layer needs oxygen. When a glass protection plate was sealed over the front of the R.S.S., high dark current resulted from the heat-up process. Therefore, a glass plate, with shims along two edges, was spaced in a distance of about 3mm (1/8") above the ZnO layer. The other two opposite edges were left open, to enable air to flow across the ZnO layer during the erasure process. Later a 1.6mm (1/16") or 3.2mm (1/8") thick Micarta sheet was substituted for the glass plate,

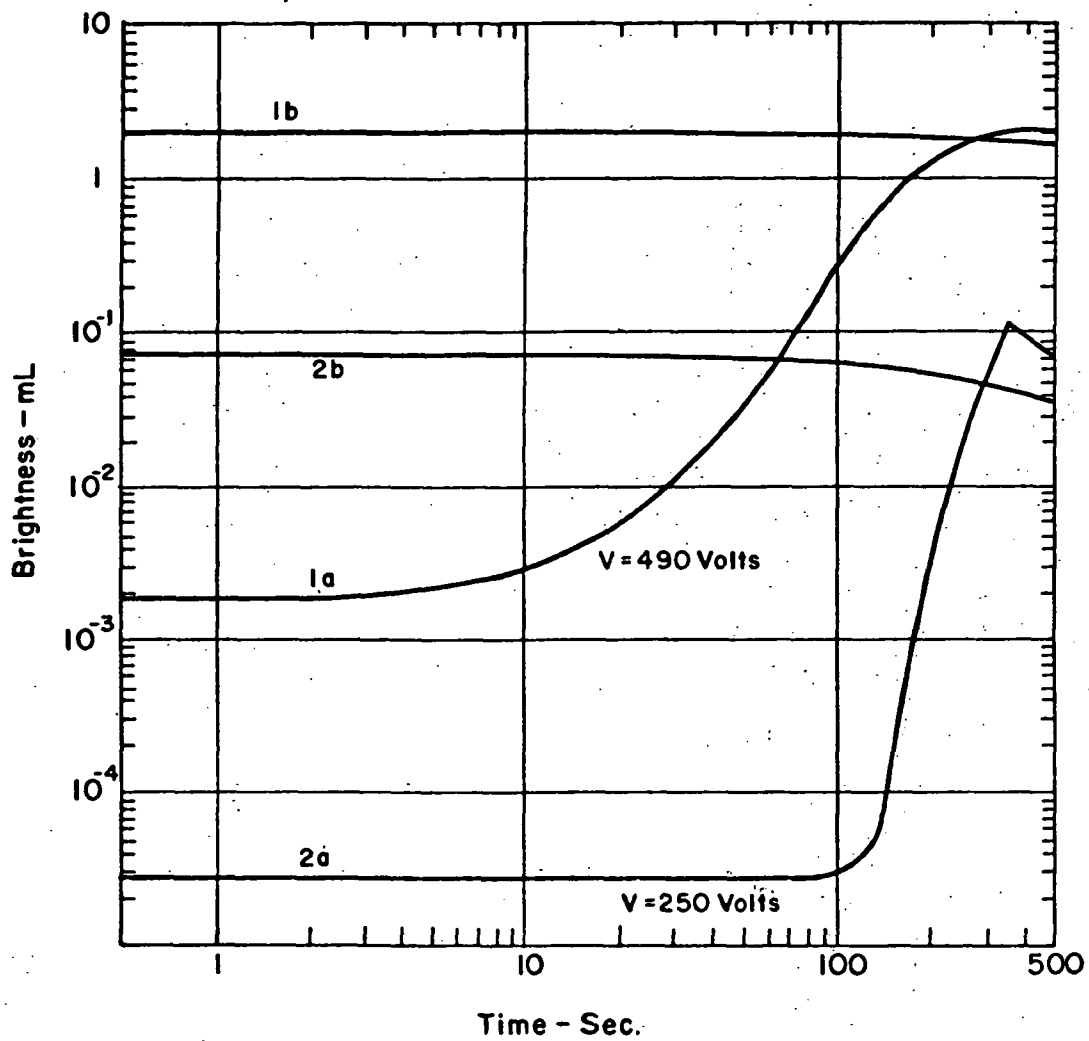


Figure 7. Characteristic Curves of a R.S.S. with Different Voltages

because its x-ray absorption was lower than that of a 1.6mm (1/16") Pyrex glass plate; and, thereby, the speed of the R.S.S. was increased. Table 1 shows brightness measurements after the storage screen was exposed through different cover plates with a given dose of x-rays at 45 kV without filter and with a 6mm (0.25") thick aluminum filter. Very high differences were observed between Pyrex glass and Micarta plate with unfiltered radiation consisting of low energy photons. Other glass plates, such as window-glass and alkali-free (Corning type 7059) glass absorbed still stronger than Pyrex glass.

Cover Plate	No Filter 2mA min Exposure		6mm Al Filter 40mA min Exposure	
	Brightness-mL	% of Brightness with Coverplate	Brightness-mL	% of Brightness with Coverplate
No cover	0.65		0.45	
1.6mm Micarta	0.45	69	0.42	93
3.2mm Micarta	0.27	41.5	0.37	82
1.6mm Pyrex Type 7740	0.03	4.6	0.15	33

Table 1. Absorption of Cover Plates With Unfiltered and Filtered 45 kV X-rays

The exposure time for a  $1.08 \times 10^{-4}$  Lambert (0.1 fL) output brightness with 45 kV 2mA in a distance of 91cm (36") was 10 to 20 sec. Since the intensity changes as the square of the reciprocal distance and roughly linearly with the current, the necessary exposure time with 45 kV (without filters) 1mA in a distance of Dcm is given by the following formula, when an average of 15 sec was taken for the 2mA x-ray exposure:

$$t = 15 \frac{D^2}{91^2} \times \frac{2}{1} \text{ sec.} = 3.7 \times 10^{-3} \frac{D^2}{1} \text{ sec.}$$

Since the intensity of the 45 kV, 2mA tube in 91cm distance is about 7R/min.,  $\frac{15}{60} \times 7 = 1.74R$  was the dose for the exposure discussed above.

Figure 8 shows the exposure time - current product and the dose as a function of the aluminum thickness with output brightness as a parameter measured with 45 kV x-ray tube voltage and 91cm (36") source-panel distance.

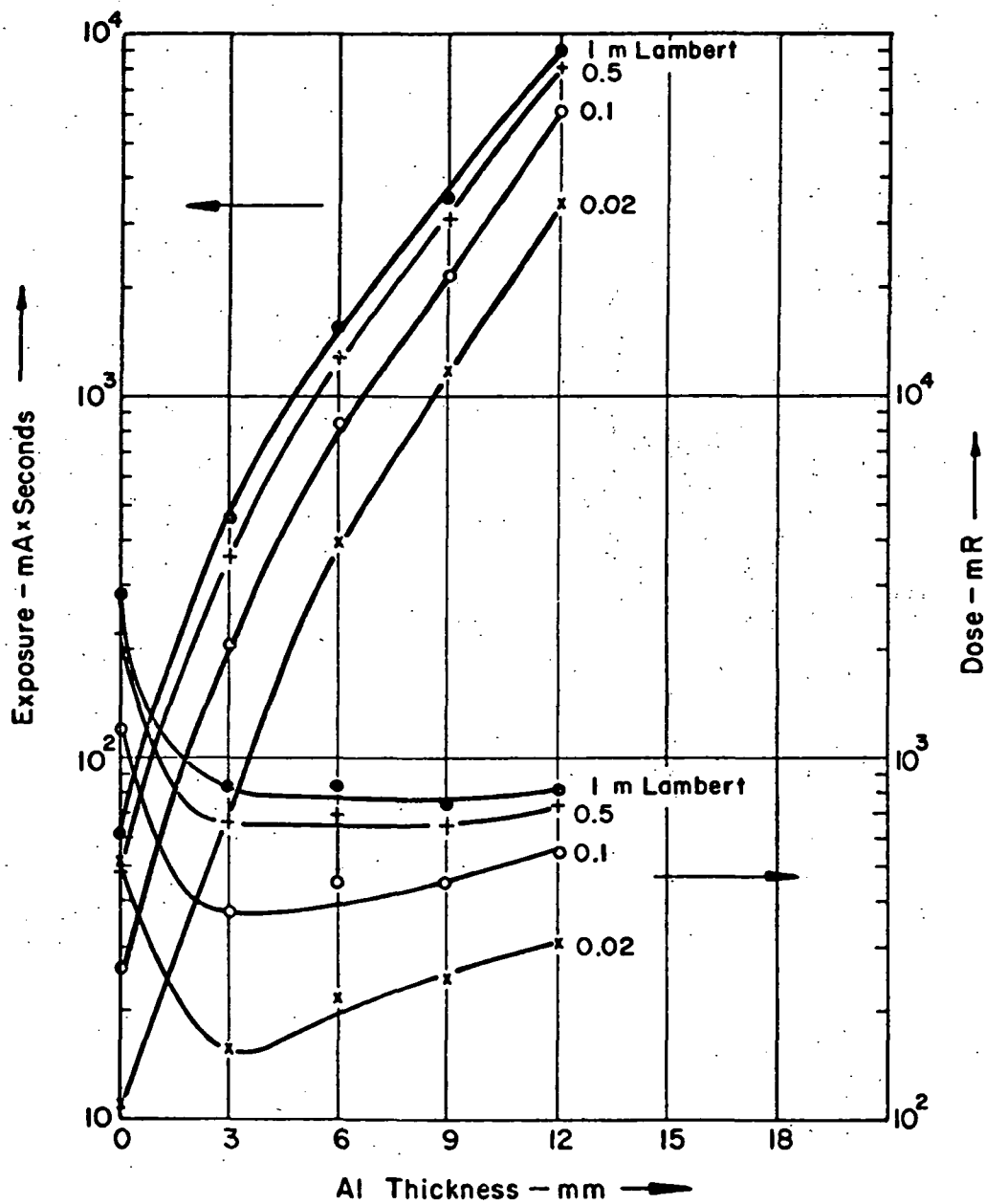


Figure 8. Exposure and Dose vs. Aluminum Thickness for Four Different Brightnesses.  
 X-ray: 45 kV, 10 mA, D = 91 cm.  
 Voltage set for Background Brightness of  $5 \times 10^{-3}$  mL ( $\approx 5 \times 10^{-3}$  fL)

We can see from these curves that the dose starts to diminish strongly with filtering. This indicates that the speed of the R.S.S. increases with increasing x-ray photon energy.

Figure 9 shows the exposure time-current product as a function of the voltage on the x-ray tube for reaching  $1.08 \times 10^{-4}$  Lamberts (0.1 fL) brightness on the panel. One of the curves give the current-time product for unfiltered, the other for filtered (6mm aluminum) radiation. The dotted lines give the x-ray intensities as a function of the voltage, both without and with the 6mm aluminum filter. The source-panel distance was 91cm in these measurements.

Table 2 gives comparison data on the speed of the R.S.S. and two popular radiographic films. A heavily filtered (3mm copper) x-ray radiation of about 100 kV was used for these data, according to American National Standard PH 28-1964. In the table approximate doses are given which produce certain densities on radiographic films<sup>(7)</sup>, and the dose needed to reach  $1.08 \times 10^{-4}$  Lamberts (0.1 fL) output brightness on the R.S.S., where the gamma is high enough to detect 2T hole on the 2% penetrameter.

Recording Medium	Required Dose in Röntgens		
	to $\gamma = 2$	to $\gamma = 3$	to 0.1 fL where $\gamma > 3$
Kodak Type M film with single lead foil	0.56	0.85	
Kodak Type R film with single lead foil	2.50	3.50	
R.S.S.			1.2

Table 2. Speed Data on Radiographic Films and the R.S.S.

In most of the measurements, the driving voltage was set so that the background brightness of the panel was about  $5.4 \times 10^{-6}$  Lamberts ( $5 \times 10^{-3}$  fL). This setting depended on the thoroughness of the erasing. When the erasing was complete, higher voltage setting resulted. However, for high contrast sensitivity, a lower voltage was better, as Figure 7 shows. To obtain the 2-2T quality level, about 2/3 of the standard voltage setting was satisfactory, and the dose had to be increased by about a factor of two.

To visually detect the 2T hole of the 2% penetrameter on a 6mm (1/4") thick Aluminum using 45 kV lightly filtered (3mm Al) x-rays (10 mA and 91cm distance) about 5 minute exposure time was needed, 5.5R falling on the sample. The dose on the R.S.S. itself was less than 1R.

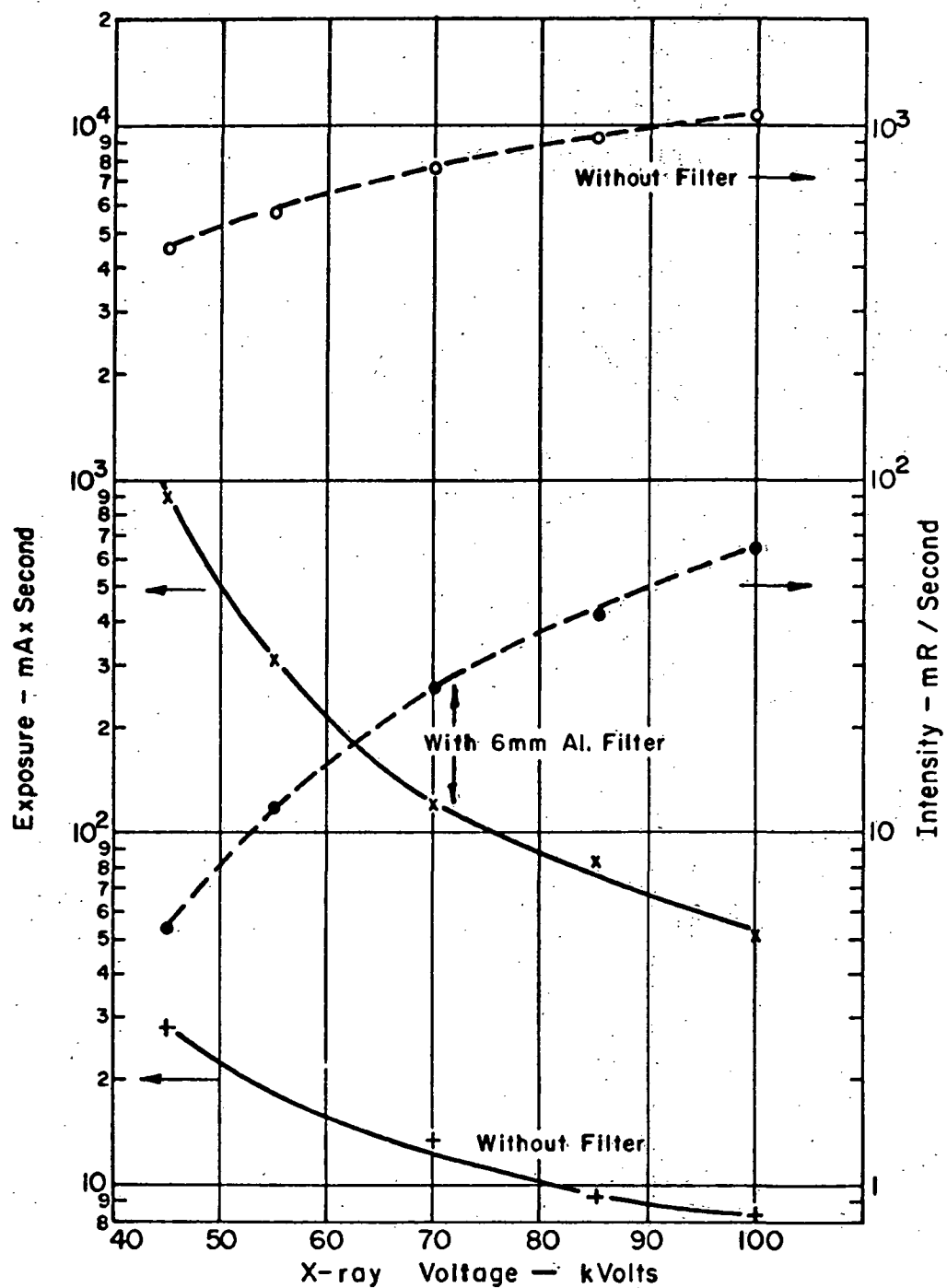


Figure 9. X-ray Exposure and Intensity vs. X-ray Tube Voltage Without Filter and With a 6 mm (0.25") Aluminum Filter

As it was mentioned, the R.S.S. can not be sealed. However, humidity is harmful for the EL layer. Therefore, it is important to bake out the panel immediately before using it, to drive out the absorbed humidity. In spite of this, if high voltage and current heats up the panel in humid atmosphere, the EL layer starts to darken visibly and a non-erasable image can be noticed on the EL layer (even without voltage).

The remedy for this deterioration would be to seal the panel in dry air or oxygen atmosphere. As an alternative and more preferable possibility would be to find a solid material, which in contact with the ZnO layer, could deliver the necessary oxygen during the erase process. Having found this material, the panel could be epoxy sealed with a cover glass, which would give protection against humidity and insure longer life for the EL layer. Time was not available for solving this problem.

Figure 10 is a photograph of a R.S.S. with its power supply. The screen shows a radiographic image of an image intensifier tube, taken about 10 minutes before the photographic exposure. The power supply contains the driving and erasing voltages.

On Figure 11 the radiographic image of an interval timer is displayed on the R.S.S. and photographed with the timer itself.

### 5.3 Radiographic Amplifier Screen (R.A.S.)

A small effort was directed toward the improvement of the radiographic amplifier screen. The goal was to increase the absolute sensitivity and the contrast sensitivity of the panel. The threshold sensitivity of the previously developed R.A.S. was about 60 mR/min. The quality level of the best panel on 6mm (0.25") aluminum was 8-2T (resolution of a 1mm (0.04") diameter hole in a 0.5mm (0.02") thick penetrometer). On these panels a 50% CdSe 50% CdS PC layer was used.

#### 5.3.1 Experiments for Improved Contrast and Gain

The conclusion from the results of the previous part of this program was that increased amount of the CdS mixed in the CdSe increased the contrast sensitivity, but slowed down the response and decay time of the panel. It was thought that substituting the CdS with some other material could also result in an increased contrast, without decreasing the speed of response.

A mixture of CdSe and ZnS was tried, but did not show any improvement. Even the spectral response of the mixture layer was the same as that of a CdSe layer.

Another series of experiments was carried out with CdSe-CdS mixture in 9:1 proportion. Sintering in nitrogen atmosphere was considered for improved characteristics. However, previous experiments in N<sub>2</sub> atmosphere showed too high dark currents. It was assumed that the cause of the high dark currents is the high concentration of Se and S vacancies, which are not



Figure 10. Photograph of a R.S.S. with Power Supply  
for Driving and Erasing Voltages



Figure 11. R.S.S. Displaying Radiographic Image of  
Timer Shown Beside Panel

5.  
compensated by oxygen acceptors, unlike in an air-baking. To eliminate the high dark currents, Se enriched CdSe-CdS mixtures were made. Sintering this mixture in N<sub>2</sub> atmosphere still did not result in the necessary characteristics. But sintering in air atmosphere at about 535°C gave high sensitivity, good reproducibility and some improvement in contrast. 0.4% Se gave the best characteristics; however, the speed of these amplifier panels was in the order of seconds.

### 5.3.2 Characteristics of R.A.S.

Several 3"x3" size radiographic amplifier screens were fabricated with transfer characteristics shown on Figure 12. The threshold sensitivity of these panels was an order of magnitude improved. It was 2 to 6 mR/min. The quality level of the best panels on 6 mm (0.25") aluminum was 4-4T (detection of 1 mm (0.04") diameter holes on 0.25 mm (0.01") thick penetrometer).

Comparing the characteristics of these R.A.S. to those of B. C. Johnson<sup>(6)</sup>, it was found that they are superior in picture quality and contrast, and equivalent in absolute sensitivity.

After the preparation method for the 3"x3" size panels had been optimized, 9"x10" panels were made. The change in size brought in some changes in the characteristics, as it was expected; and a large number of panels had to be prepared, until all the characteristics were acceptable. The most disturbing fault was a mottled picture structure at low level illumination, which improved at increased brightnesses.

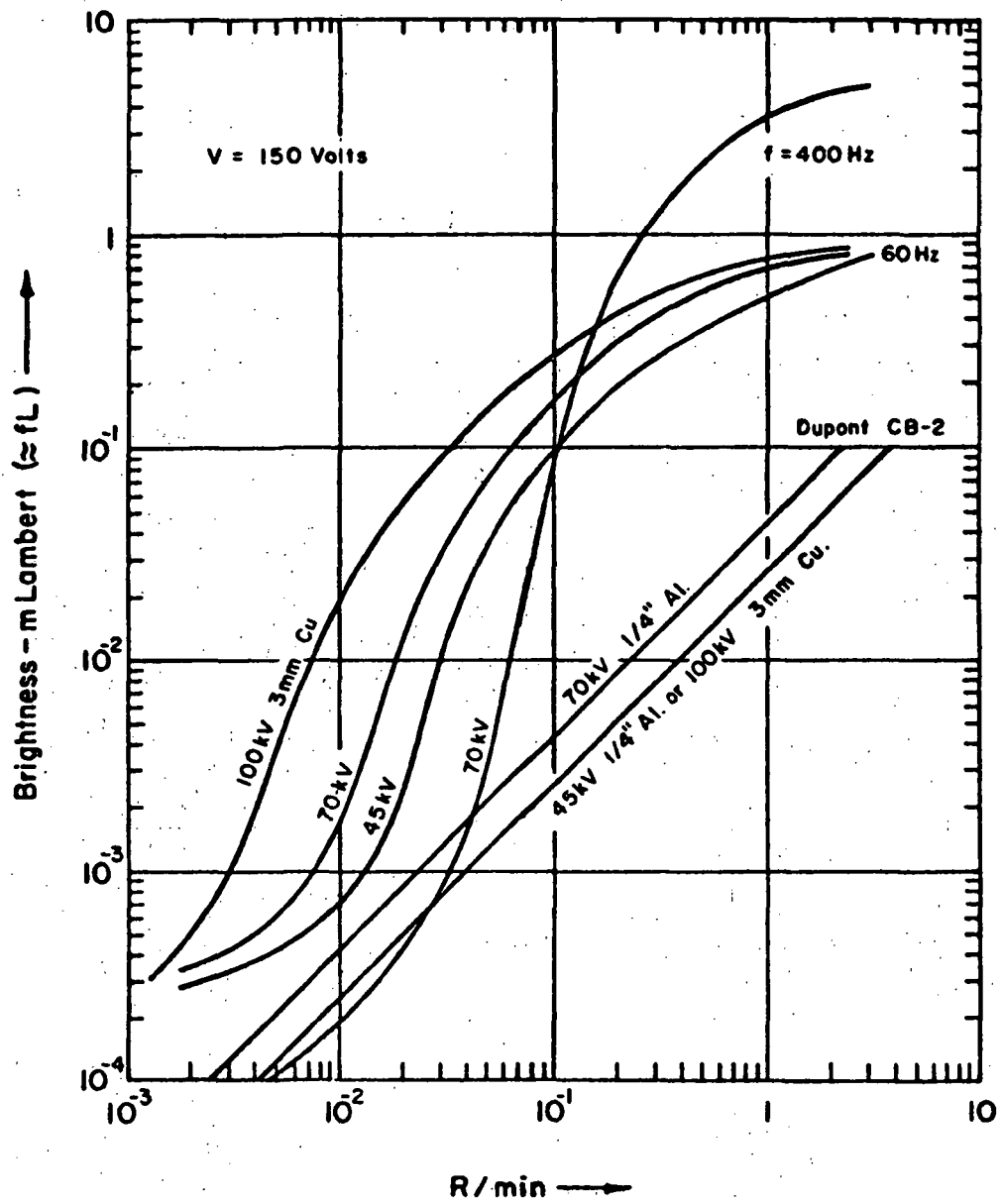


Figure 12. Transfer Characteristics of a R.A.S.

## SECTION 6

### DISCUSSION

The main effort of this program was directed toward the development of a solid state radiographic storage screen which could be used in radiographic non-destructive testing and would substitute for photographic film. Consequently, the requirements were: (1) 2-2T quality level as specified in MIL-STD-453; (2) speed comparable to Kodak type M radiographic film; (3) resolution 200 lines per inch or better; (4) storage time without deterioration of the image: more than five minutes; (5) erasure time not more than a few minutes.

The PC-EL sandwich type storage panel, with ZnO as the radiographic sensing element (which was developed in the previous program), nearly satisfied all these requirements, excepting the first. Therefore, the main goal of the program reported here was to improve the contrast sensitivity of the ZnO panel or develop another radiographic storage system which could meet the specifications.

Several possible approaches were considered, and experiments started in various directions. However, it was found that the simplest and most promising path was to improve the contrast of the ZnO by appropriate preparation of the powder.

The work in this direction was successful: Radiographic storage screens were fabricated, satisfying most of the requirements for industrial non-destructive testing.

The R.S.S. is a solid state X-ray to visible image converter. It is reusable and can serve as an inexpensive replacement for expensive X-ray photographic film in industrial applications, where record keeping is not needed. Permanent records may, however, be obtained conveniently by photographing the screen image with a photographic camera (with Polaroid or inexpensive film) or by using a video camera with records maintained on video tape.

Upon exposure, the radiographic image can be seen immediately without processing. The displayed image is stored, without excessive deterioration, for 10 to 60 minutes, depending on the preparation of the screen and on working parameters. Much longer storage times can be obtained by disconnecting the electric field from the panel.

The absolute sensitivity (speed) of the R.S.S. is equivalent to high resolution, high contrast X-ray films. It has a resolution of about 8 line pairs/mm (400 TV lines/inch). Its contrast (gamma) depends on the driving voltage and X-ray dose and can be set between  $\gamma = 2$  to  $\gamma = 7$ . Practically, the radiographic quality level reached is 2-2T.

Before taking a radiographic image with the R.S.S., the previous image has to be erased by heating, which can conveniently be accomplished electrically, connecting the two lead wires on the two borders of the tin oxide coated glass substrate to about 50 volts (for 20 cm x 25 cm screen). Three to four minute heating and about five minute cooling prepares the screen for a new exposure.

The time loss caused by the erasing process can be eliminated by using two or three R.S.S. alternatively.

Westinghouse has introduced the following three sizes of these panels for commercial sale:

7.5 cm x 7.5 cm	(3" x 3")	Type No.: WX-31980
15 cm x 20 cm	(6" x 8")	Type No.: WX-31981
20 cm x 25 cm	(8" x 10")	Type No.: WX-31982

A smaller effort was devoted in this program to the improvement of the radiographic non-storage type amplifier panel, known as the radiographic amplifier screen (R.A.S.). Increased absolute sensitivity by a decade and some improvement in the contrast sensitivity were achieved.

The R.A.S. is an X-ray to visible image converter. It is a replacement for fluoroscopic screen or X-ray intensifier tube, offering:

- (1) Three times higher resolution than the fluoroscopic screen or intensifier tube.
- (2) Two to six times higher contrast than the fluoroscopic screen or intensifier tube.
- (3) Ten to fifty times higher brightness than the fluoroscopic screen (Dupont CB-2).

Westinghouse has introduced the following three sizes of the R.A.S. for commercial sale:

25 cm x 25 cm	(10" x 10")	Type No.: WX-31661
12.5 cm x 12.5 cm	(5" x 5")	Type No.: WX-31662
7.5 cm x 7.5 cm	(3" x 3")	Type No.: WX-31663

## SECTION 7

### RECOMMENDATIONS

The developed radiographic screens are suitable for many practical applications, mostly in radiographic non-destructive testing and in special medical applications.

The radiographic amplifier screen is of sealed construction. It is protected against humidity and can be used in vacuum; consequently, its application in space experiments could be possible.

The radiographic storage screen is not sealed, because it needs oxygen for the erasing process. Consequently, its EL layer deteriorates faster in humid atmosphere, and it can not be used in vacuum.

For increasing the life of the R.S.S. and to make its use possible in space experiments, it is proposed to develop a simple sealing method, either by closing the panel in a dry air atmosphere or by the application of a solid material (a thin sheet in contact with the ZnO layer) which could release oxygen in the erasing process.

For practical applications, it would be preferable to build the R.S.S. on a flexible plastic substrate instead of glass. Principally, there are no difficulties to doing this and a small effort for the development of the fabrication technique would result in a simple and less expensive plastic construction.

Therefore, it is recommended to support a small work program for the development of plastic radiographic storage screens.

## SECTION 8

### REFERENCES

1. Z. Szepesi: Solid State Radiographic Image Amplifiers. Final Report, Part A, on Contract NAS8-21206. 33 pages. May 1968.  
NASA CR-102299.
2. Z. Szepesi: Solid State Radiographic Image Amplifiers. Final Report, Part B, on Contract NAS8-21206. 53 pages. November 1969.  
NASA CR-61328.
3. E. S. Lamar - S. Hecht - S. Shlaer - C. D. Hendley: Size, Shape, and Contrast in Detection of Targets by Daylight Vision I. Data and Analytical Description.  
Journ. Opt. Society Am. 37, 531-545, 1947.
4. H. Ivey et als.: Electroluminescent Phosphor with Improved Voltage Characteristics.  
Final Report, Contract No. AF 19(122)-458, Subcontract No. 105. MIT Lincoln Laboratory. November 1959.
5. R. L. Brown, Sr.: Cascaded Solid-State Image Amplifier Panels.  
U.S. Patent 3, 548, 214, December 15, 1970.
6. B. P. Johnson: Improvements of Solid State X-ray Image Intensifiers.  
Phys. Med. Biol. 15, 343-348, 1970.
7. Eastman Kodak Company: Kodak Films for Industrial Radiography. Supplement to Radiography in Modern Industry.

## SECTION 9

## APPEND IX

## APPENDIX A

### FABRICATION PROCEDURE OF RADIOGRAPHIC STORAGE SCREENS

#### I. Preparation of ZnO Powders

1. Weigh 80g pure ZnO powder and mix 100mg  $\text{NaSO}_4$  and 60mg  $\text{PbCl}_2$  with it, and load it into an open quartz boat.
2. Bake the mixture in a furnace with slowly flowing air (2 CFH) at  $950^\circ\text{C}$  ( $1742^\circ\text{F}$ ) for two hours.
3. After the powder cools down, place it in a Waring blender 1/3 full with deionized water and blend it for 1 minute at high speed.
4. Pour mixture into 1 liter Erlenmyer flask; let the powder settle for 5 minutes and decant the water.
5. Repeat rinsing, settling (10 minutes) and decanting two other times with deionized water.
6. Rinse with 2-propanol, settle (10 minutes) and decant.
7. Rinse again with 2-propanol; ultrasonically agitate for 3 minutes.
8. Filter through Millipore filter (type AAWP4700-AA-0.8 $\mu$ ).
9. Dry in forced air oven at  $135^\circ\text{C}$  ( $275^\circ\text{F}$ ) for 30 minutes.
10. Shake powder through 200 mesh sieve.

#### II. Composition of ZnO Mixtures for Radiographic Amplifiers

##### 1. Mix:

72g ZnO powder (prepared as described above)  
→ 12g silicone resin DC-804 (60% solid) (See Addendum, Section 3);  
23g Iso-Amyl Alcohol  
2g Butyl Carbitol

2. Ball mill the mixture with 25 3/8" diameter steel balls in a 4 fluid oz. glass bottle for 45 minutes.
3. After taking out the balls, agitate the mixture for about 2 minutes with ultrasonic agitation and use it for blading.

### III. Fabrication of the Panel

1. Etch a 5/16" wide strip from the tin-oxide coating on two opposite edges of the substrate glass (Pyrex, Corning Glass type 4470).
2. Clean substrate glass plate by washing in Alconox detergent, wiping with  $\text{CaCO}_3$  water paste, rinse in deionized water, and vapor degrease and dry in 2-propanol vapor.
3. Bake in a 500°C furnace for 15 minutes.
4. Spray an EL layer on the substrate as described in Appendix C of Reference 2, Schedule 1-4, using mixture 1a.
5. Blade the ZnO mixture (specified in paragraph 11 above) on top of the EL layer with Shandon blading machine with thickness setting of 17.5 mils.
6. Cover the bladed layer and let it dry slowly (at least 24 hours) at room temperature.
7. Bake the panel for 90 minutes at 130°C (265°F).
8. Paint 1 layer Ucilon\* 15% on 2 opposite edges where tin-oxide was etched out on top of the ZnO layer.
9. Bake the panel for 30 minutes at 100°C.
10. Apply Emerson Cummings V-91 silver epoxy on top of the two Ucilon coated edges.
11. Bake 30 minutes at 100°C (212°F).
12. Evaporate a PbO + Au electrode on top of the ZnO layer until the monitoring slide shows about 3 ohms/square.
13. Prepare two 10" long, 18 gage copper wire with 2 feet flexible 20 gage insulated stranded wire soldered to one end of each.
14. Clean two opposing edges of the glass plate (other than the one with the silver epoxy) and cement the copper wire all along both edges with Emerson-Cummings V-91 silver epoxy. These wires serve for heating the tin-oxide electrode to erase the stored image.
15. Connect the two top electrodes (see item 10) with No. 26 stranded insulated wire and attach a 2-feet long lead from the same wire with Emerson-Cummings V-91 silver epoxy. This lead serves for the connection of the driving voltage to the top Au electrode.
16. Paint about a 5/8" wide border on the four edges of the glass substrate (not coated side) with black paint.

\*Made by M&T Chemicals, Inc., Rahway, New Jersey

17. Paint a 1.5 mm (0.06") thick Micarta cover-plate, the same size as the glass substrate, with black spray enamel.
18. Mount the cover plate above the panel with a spacer of about 6 mm (1/4"), leaving two ends open for free air circulation.

## APPENDIX B

### FABRICATION PROCEDURE OF RADIOGRAPHIC AMPLIFIER SCREENS

#### 1. Panel Fabrication

##### 1. Substrate Preparation:

- a. Clean tin-oxide coated Pyrex (Corning type 4470) glass plate as described in Appendix F, Reference 2.
  - b. Platinum coat center two-thirds of two opposing edges.
  - c. Fire platinum coat at 525°C (977°F) - 1 hour in air.
2. Preparation of CdSe-CdS powder. See Part II below.
  3. Settling of the PC powder. See Part III below.
  4. Sintering. See Part IV below.
  5. Evaporate 750mg CdSe at 18 inch distance in high vacuum on top of the sintered CdSe layer.
  6. Spray 2 layers of 5% Ucilon White\* Type 400-9.
  7. Spray 2 layers of 5% Ucilon Grey\* Type 400-10, containing 3% (of solid plastic) lamp black.
  8. Bake in forced air oven at 135°C (275°F) for 30 minutes.
  9. Brush 2 layers of 25% Ucilon White on 4 edges.
  10. Repeat step 8.
  11. Spray 2 layers of TiO<sub>2</sub> spray mixture. See Part V below.
  12. Spray 6 layers of green EL phosphor, Westinghouse type VB-242P, and 3 layers of clear coat (steps 1 to 6 of Appendix C, Reference 2) using mixtures 1 then 2.
  13. Brush 3 layers of Ucilon on 4 edges only.
  14. Bake at 135°C (275°F) for 30 minutes in forced air oven.
  15. Apply Emerson-Cummings V-91 silver epoxy with rubber pad applicator to the two opposing edges not covered with platinum. This will connect to the evaporated gold layer.
  16. Bake at 135°C (275°F) for 15 minutes.
  17. Evaporate top transparent gold electrode PbO + Au (step 7 of Appendix C, Reference 2).

\*Made by M & T Chemicals, Inc., Rahway, New Jersey

18. Pretest.
  - a. Sensitivity.
  - b. Time response
  - c. Imperfections, spots, bright edges.
19. Cover electrode edges on substrates with 1/16 inch wide masking tape to protect electrode from epoxy.
20. Spray several coats of Krylon crystal clear spray coating, Type 1302, on top of gold layer.
21. Bake panel for 30 minutes at 80°C (176°F).
22. Attach wires to two electrodes (one to tin-oxide, other to gold) with Emerson-Cummings V-91 silver epoxy.
23. Apply about 50g (for 9" x 10" panels) of Emerson-Cummings No. 1266 epoxy to the center of the coated substrate.
24. Place a precisely cut cover glass against the epoxy on top of substrate and carefully align.
25. Wipe excess epoxy from edges as it squeezes out and apply a flat steel plate on top of cover glass as a weight, heavy enough to hold cover glass parallel and near the panel during cure.
26. Allow to cure at room temperature for 16 hours and remove excess epoxy with razor blade, taking care not to damage electrodes.
27. Remove masking tape from edges.

#### 11. Mixing and Prebaking the CdSe-CdS Powder

1. Weigh 90 grams of CdSe and 10 grams CdS powder (G.E. electronic grade) and mix it in a Pyrex beaker.
2. Place the well mixed powder in a quartz boat and heat it slowly in nitrogen atmosphere until the temperature reaches 1075°C (1966°F). Bake it for 30 minutes at this temperature and cool down still in nitrogen atmosphere.
3. Grind the material in diamonite mortar.
4. Place this material in a beaker and mix to it 400 mg of high purity selenium powder.
5. Mix a 1% water solution (be weight) of  $\text{CuCl}_2 + 2 \text{H}_2\text{O}$  (Fisher Certified), and add 2.5 ml to the CdSe-CdS-Se mixture.
6. Bake this mixture at 135°C for one hour.

7. Weigh 1 gram of dry  $\text{CdCl}_2$  and 500 mg  $\text{NH}_4\text{Cl}$  and add to the powder mixture.
8. Prebake the powder mixture at  $540^\circ\text{C}$  ( $1004^\circ\text{F}$ ) for 90 minutes in a quartz dish with a cover in nitrogen atmosphere (5 CFH).
9. After cooling, grind the material in a diamonite mortar.

### III. Settling

1. Weigh about 68 grams of the prepared CdSe-CdS powder, transfer it to a ceramic ball milling jar; mix about 100 ml Xylene with it and ball mill for nineteen hours.
2. Place the substrate glass plates in a perfectly horizontal plane at the bottom of a 12" x 12" glass jar.
3. Fill the jar with a 1.0% ethyl cellulose Xylene solution (filtered through a  $8\mu\text{m}$  size Millipore filter) to about 4" height above the substrate glasses.
4. Pour the ball milled PC mixture in the settling jar and let it settle until the Xylene clears up (about 2 hours).
5. Siphon off cushion and let panel dry in tank 10 to 16 hours.
6. Remove panel from tank and transfer to forced air oven and bake at  $80^\circ\text{C}$  ( $176^\circ\text{F}$ ) for half an hour. Increase the temperature to  $135^\circ\text{C}$  ( $275^\circ\text{F}$ ) and bake for an additional half hour.

### IV. Sintering

1. Place panel on a Vycor plate and cover with a pyrex dish.
2. Bake in a furnace of  $535^\circ\text{C}$  ( $995^\circ\text{F}$ ) for 60 minutes (air atmosphere).
3. Turn the Vycor plate with the panel  $180^\circ$  and bake an additional 20 minutes.
4. Remove panel from furnace and allow to cool under a strong airflow on the pyrex dish.
5. Turn down the temperature of the furnace to  $515^\circ\text{C}$  ( $959^\circ\text{F}$ ) and bake the panel without cover for 15 minutes at this temperature.
6. Place the panel in a  $190^\circ\text{C}$  ( $374^\circ\text{F}$ ) furnace (air atmosphere) and age for 16 hours.
7. Cool the panel under room light.

V. Titanium Dioxide Spray Mixture

45g fine  $\text{TiO}_2$  powder

90 ml 5% solution of cyanoethyl starch (CE)\*

90 ml 5% solution of cyanoethyl sucrose (CES)\*

\*Sold by Eastman Chemical Corporation

## APPENDIX C

### OPERATIONAL PROCEDURE FOR RADIOGRAPHIC

#### STORAGE SCREENS

The R.S.S. is not protected against humidity, which is harmful to the EL layer, when current is driven through it. The higher the current (higher brightness) the faster the deterioration. The micarta cover plate gives protection only against mechanical injuries and leaves free air circulation to the ZnO layer, which needs oxygen for the erasure.

Before using the storage screen, it must be heated to about 100°C to 130°C and then cooled. The heating has a double effect: (1) erases the previous image; (2) drives out the humidity from the EL layer, thereby increasing the life of the panel.

The heating can be accomplished by three methods.

1. Bake the panel in a furnace.
2. Irradiate it with an infrared lamp.
3. Heat electrically the tin oxide coating of the substrate glass.

The third method is the most convenient. The delivered panels have two leads for the electric heating. Connecting about 50 volts AC or DC to them, the tin oxide coating will heat up to about 130°C during 3 to 4 minutes. This is sufficient for erasing the image.

After heating, the panel should be allowed to cool down to room temperature. For highest contrast sensitivity, a fast cooling is advisable. Placing the panel, its output side down, on a flat metal sheet and blowing air through the gap between the glass and cover plate is recommended.

During exposure to X-rays, the panel does not need driving voltage, but it can be applied if one wants to see the development of the image. For high speed (high absolute sensitivity), the voltage should be as high as possible, and the frequency low. The maximum voltage should not be higher than about 600 volts. It is good practice to determine the useful voltage before exposure by increasing the voltage until a faint background light starts to be visible on the panel.

For high contrast, lower voltage (about 50 to 65% of the maximum) should be used. In this case, the exposure time has to be increased by a factor of about two.

Some storage screens, having very long storage times, do not erase completely with the proposed baking process. Exposing the panel uniformly with a high X-ray dose helps to achieve complete erasing.

Visible image pattern on the EL layer without voltage indicates that the EL layer deteriorated, caused by high current - high brightness in humid atmosphere. This image can not be erased.

It would be advantageous to store the storage panel in dry atmosphere. But the most important would be to protect it from humid atmosphere during the time the image is displayed (voltage connected on).

The time loss, caused by the erasing process, can be overcome by using 2 or 3 panels successively.

**SOLID STATE RADIOGRAPHIC IMAGE AMPLIFIERS**

**ADDENDUM TO FINAL REPORT, PART C**

**Covering the Period of July 1, 1971 to February 29, 1972**

**Report Written by: Zoltan Szepesi**

**March 1972**

**Prepared for:**

**George C. Marshall Space Flight Center  
National Aeronautics and Space Administration  
Huntsville, Alabama**

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**ELMIRA, NEW YORK**

## INTRODUCTION

A time extension of eight months was granted on this contract for solving some problems which presented itself toward the end of the working period. These problems were (1) breakdown of the X-ray generators; (2) spottiness of the tin-oxide coated glass plates; and (3) cracks developed in the delivered radiographic storage screens.

This Addendum will discuss the above mentioned problems.

## SECTION 1

### X-RAY GENERATOR

The X-ray generator has broken down before the characteristics of the radiographic panels, which had to be delivered to the Marshall Space Flight Center (MSFC), have been measured. It was found that the X-ray tube became gassy, probably from an electric discharge in the tube. Replacing the tube and the cable restored the generator.

## SECTION 2

### GLASS SUBSTRATES

The tin-oxide coated Pyrex glass substrates, used in the fabrication of both types of radiographic converter screens, were very often full of smaller or larger spots, fingerprints, pinholes, etc. Often the tin-oxide coating was non-uniform on them. These faults influenced largely the sensitization process of the PC layer in the radiographic amplifier screen, which was baked at about 530°C, resulting in spotty, grainy and non-uniform structure of the displayed image.

The last acquired batch of such glass plates (IRR glass made by Corning Glass Works) was full of small cracks, caused by the polishing of the glass. Deep little holes were etched into the glass, often filled with the polishing rouge. This spotty structure of the glass surface caused very disturbing grainy structure of the displayed image in the finished amplifier screen.

Evaporating a thin film of SiO seemed to improve the graininess in a first experiment, but repeated experiments did not confirm this.

The proper polishing and specially careful coating of a Pyrex plate would have been too expensive (a quotation of \$362.12 each, in quantities of 10 to 25 pieces was received), and could not be considered.

Instead of the Pyrex glass, an alkali free, type No. 7059, glass was proposed, which is available in less than 1.5 mm (about 1/16") thickness with smooth surface and can be tin-oxide coated without polishing. However, the X-ray absorption of this glass was much higher than that of the Pyrex glass and was not acceptable for lower energy X-rays.

Another Corning glass, the "Chemcor" (Type 0313), had about the same absorption coefficient than the Pyrex glass. However, samples were not available with appropriate tin oxide coating and no experiments were made with it.

The best image structure was obtained on tin oxide coated "Tempax" glass plates, acquired from Schott Optical Glass, Inc. Though there were some fingerprints and other, larger nonuniformities on the first batch delivered to us, these faults will probably be eliminated in the next shipment and it is expected that image intensifier panels with improved graininess and picture quality will be fabricated with higher yield.

One 25 cm x 25 cm (about 10" x 10") size radiographic amplifier screen built on a 3mm (about 1/8") thick Tempax glass was delivered to the MSFC.

### SECTION 3

#### STORAGE SCREEN DEFECTS

About half of the storage panels, shipped to the MSFC, developed cracks in the ZnO layer within a few weeks after the delivery in Huntsville. Although little, and not very disturbing cracks were noticed in a small number of panels in Elmira during more than a year working period, no extensive cracking as shown on the panels in Huntsville, was observed. The explanation of this fact could be that the cracking occurs preferably in higher humidity.

Since the ZnO powder was embedded in a Dow-Corning 804 silicone resin, which has poor flexibility, it was thought that by using a more flexible resin, the cracking should be eliminated.

The DC-808 silicone resin has excellent flexibility and its craze life at 250°C is 5000 hours compared to the craze life of 100 hours of the DC-804 resin. Mixing the two resins, should result also in appropriate characteristics.

Experiments were made with the DC-808 alone and with a mixture of DC-808 and DC-804 in 1 to 1 and in 1 to 3 proportions. The panels made with the DC-808 did not show as good contrast as those made with the DC-804 resin. Those, made with the mixture, though somewhat less contrasty, still were acceptable (have 2-2T quality level). Life tests, carried out during several months on these panels did not show any deterioration of the ZnO layer.

One storage panel made with 1 to 1 ratio resin mixture and another made with 1 to 3 ratio were delivered to the MSFC.

Another defect, reported on two storage panels which were delivered first, was a short circuit between the tin oxide coating and the top gold electrode on the edge of the panel. This defect was eliminated on the panels which followed the two first, by etching out a narrow band of the tin oxide coating at the edge, where the top electrode connecting lead was attached.

Life tests are being continued, but it is thought that no failure will be developed. However, a slow deterioration and decreased brightness of the EL layer is to be expected.